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Chemical Analysis of Ore Tailings

Reference Materials

Group II

Final Report

by

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MICROMEDIA

As part of Canadian Certified Reference Materials Project, 24 samples (six bottles each of four materials) were received at Peterborough on 28 December 1987. These samples were analyzed for the parameters of Group #2, namely total sulphur, sulphate-sulphur, carbonate (as CO_2) and moisture. The methods used were as follows:-

1) Total sulphur was determined by wet oxidation of the sample with nitric acid and bromine (used in solution CCl_4), followed by gravimetric determination of sulphur as barium sulphate. Details of the procedure may be found in Ref. (1).

2) Sulphate-sulphur was liberated from insoluble sulphates by digestion with sodium carbonate solution, followed by gravimetric determination of sulphate as barium sulphate (2).

3) Carbonate (reported as CO_2) was determined by decomposition of the samples by acid, and gravimetric determination of the evolved CO_2. The procedure in Ref. (3) was followed with minor modification as noted below.

4) Moisture was determined by loss on drying (L.O.D.) in air at 103 - 105°C for 1 hour (4). Further drying caused loss of other constituents, as noted below.

Results and Comments

The results of the analyses are summarized in Table 1. Some problems were encountered with certain of the samples. Those high in total sulphur (RTS-2, 3 & 4, es-

pecially the last) caused difficulties in the CO_2 determination, as the qualities of sulphurous gases evolved were more than the protective absorbers could handle, and very high and variable results were obtained until the sample sizes were much reduced. It was found necessary to add hydrogen peroxide directly to the sample in the decomposition flask with RTS-4. The small samples then gave results which were not much above the detection limit of the method as used. The same samples, in the determination of moisture by L.O.D. at 103-5° C lost nearly as much mass in the second hour of drying as in the first. It was obvious from the smell of SO_2 on opening the oven that oxidation of sulphides and consequent loss of this gas were also occurring. Cautions to the uses of these as reference materials are recommended.

The figures reported are the results of single determinations. Duplicates were carried through with representative samples, and the precision of the analysis within a bottled sample (the combined result of mixing, sub-sampling and measurement) was assessed for each procedure. These results are shown in Table 2, and should be considered as assessing the variability from bottle to bottle as reported in Table 1.

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Personal:

The work was carried out by:

Dr. R.C. Makhija - Project Manager and Principal Investigator.

Mr. J. LaPlante - Technician.

Prof. R.A. Stairs - Consultant.

Table 1

Moisture (H_2O, 105°C)

Our Sample #	Your sample #	Weight of sub-sample taken, g	% Moisture 1 hr. heating & cooling	Further 1 hr. heating
RR-83-03	RTS-1A	1.0166	1.58	1.80
-04	B	1.0002	1.44	1.76
-05	C	1.0195	1.65	1.87
-06	D	0.9956	1.57	1.88
-07	E	1.0703	1.59	1.84
-08	F	1.0374	1.53	1.90
RR-88-09	RTS-2A	0.9928	1.63	2.04
-10	-2B	1.0160	1.60	2.09
-11	-2C	0.9920	1.67	2.08
-12	-2D	1.0374	1.64	2.03
-13	-2E	1.0254	1.64	2.11
-14	-2F	1.0148	1.63	2.06
RR-88-15	RTS-3A	1.0228	1.77	2.08
-16	-3B	1.0328	1.73	2.10
-17	-3C	1.0066	1.75	2.10
-18	-3D	1.0106	1.79	2.16
-19	-3E	1.0589	1.79	2.08
-20	-3F	1.0909	1.82	2.10
RR-88-21	RTS-4A	0.9993	0.14	0.25
-22	-4B	1.0160	0.15	0.26
-23	-4C	1.0298	0.14	0.24
-24	-4D	1.0827	0.14	0.25
-25	-4E	1.0118	0.16	0.29
-26	-4F	1.0033	0.16	0.34

*Samples start to decompose and a smell of SO_2 is noticed in the room.

Sulphate - Sulphur

Our Sample #	Your Sample #	Weight of Sub-sample g	% S
RR-88-03	RTS-1A	1.0007	1.29
-04	-1B	1.0006	1.29
-05	-1C	1.0012	1.31
-06	-1D	1.0014	1.30
-07	-1E	1.0009	1.29
-08	-1F	1.0004	1.29
RR-88-09	RTS-2A	1.0008	3.92
-10	-2B	1.0000	4.00
-11	-2C	0.9995	4.00
-12	-2D	0.9985	3.90
-13	-2E	1.0014	3.88
-14	-2F	1.0009	3.92
RR-88-15	RTS-3A	1.0004	1.42
-16	-3B	1.0002	1.46
-17	-3C	1.0003	1.49
-18	-3D	1.0008	1.47
-19	-3E	1.0013	1.43
-20	-3F	1.0006	1.45
RR-88-21	RTS-4A	0.9994	0.16
-22	-4B	1.0007	0.16
-23	-4C	1.0008	0.16
-24	-4D	1.0022	0.16
-25	-4E	1.0018	0.16
-26	-4F	1.0006	0.16

Total Sulphur

Our Sample #	Your Sample #	Weight of Sub-sample taken, g	% Total Sulphur
RR-88-03	RTS-1A	0.4997	1.62
-04	-1B	0.2513	1.64
-05	-1C	0.2514	1.56
-06	-1D	0.2514	1.62
-07	-1E	0.2503	1.59
-08	-1F	0.2508	1.56
RR-88-09	RTS-2A	0.5025	19.34
-10	-2B	0.5017	19.34
-11	-2C	0.5014	19.23
-12	-2D	0.5005	19.35
-13	-2E	0.5007	19.30
-14	-2F	0.5012	19.38
RR-88-15	RTS-3A	0.5004	10.10
-16	-3B	0.5012	10.34
-17	-3C	0.5007	10.30
-18	-3D	0.5005	10.13
-19	-3E	0.5010	10.24
-20	-3F	0.5005	10.26
RR-88-21	RTS-4A	0.2509	35.89
-22	-4B	0.2527	35.91
-23	-4C	0.2512	35.67
-24	-4D	0.2536	35.93
-25	-4E	0.2525	35.99
-26	-4F	0.2520	35.99

Carbonate (CO_2)

Our Sample #	Your Sample #	Weight of Sub-sample taken, g	% CO_2
RR-88-03	RTS-1A	5.0023	0.05
-04	-1B	5.0031	0.06
-05	-1C	5.0027	0.054
-06	-1D	5.0035	0.06
-07	-1E	5.0047	0.05
-08	-1F	5.0566	0.06
RR-88-09	RTS-2A	4.9976	0.01
-10	-2B	5.0005	0.03
-11	-2C	5.0297	0.016
-12	-2D	5.0050	0.02
-13	-2E	5.0008	0.02
-14	-2F	5.0548	0.02
RR-88-15	RTS-3A	5.0474	0.06
-16	-3B	4.9995	0.06
-17	-3C	5.0637	0.07
-18	-3D	4.9979	0.09
-19	-3E	5.0016	0.09
-20	-3F	5.0646	0.07
RR-88-21	RTS-4A	0.5034	0.30
-22	-4B	0.5026	0.34
-23	-4C	0.5034	0.30
-24	-4D	0.5023	0.32
-25	-4E	0.5020	0.36
-26	-4F	0.5014	0.34

Table 2

Precision of Individual Analyses
(Absolute Standard Deviations, %)

Sample	Moisture	SO_4^{2-} - S	S total	CO_2
RTS - 1	0.04	0.02	0.01	0.01
RTS - 2	0.04	0.16	0.03	0.01
RTS - 3	0.04	0.07	0.01	0.01
RTS - 4	0.02	0.01	0.47	0.1

REFERENCES

1. Elsie M. Donaldson, "Methods for the Analysis of Ores Rocks and Related Materials", Monograph 881, 2nd ed., CANMET, Ottawa, 1982, p. 354.

2. Ibid., p. 364.

3. Ibid., p. 305.

4. Ibid., p. 328.